# RESEARCH SUMMARY

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The current primary focus of the our current work is to synthesize new and/or improved strongly correlated electron metallic oxide compounds and carry out physical property measurements on them to search for novel physical behaviors and/or ground states, and to clarify the origins of interesting properties of known materials. In the following some background in our research area and some our progress is outlined. Literature references by other groups have the prefix "L" and are listed at the end of this narrative. Our papers in this resume are referenced as either straight numerals for our papers in the list of refereed publications or preceeded by "O" for our "Other Publications". All three lists of references are given below.

## High Tc Cuprates

The discovery of superconducting transition temperatures $T_c$ up to about 35 K in what turned out to be the layered cuprate $La_{2-x}Ba_xCuO_4$ by Bednorz and Müller in 1986 [L1-L3] stimulated extraordinary worldwide experimental and theoretical research activity to understand the physics of the materials and generate new compounds. The physical properties of this and related families of materials were found to be highly anisotropic, reflecting the layered crystal structures in which the Cu atoms form a square lattice as shown in the left panel of Fig. 1 where an oxygen atom resides between each nearest-neighbor pair of copper atoms. Our discovery in early 1987 of long-range antiferromagnetic order at about 250 K in the undoped insulating parent compound $La_2CuO_4$ [92, 94, 95] demonstrated that the Cu atoms carry local magnetic moments as expected from an ionic model for the $d^9$ spin-1/2 $Cu^{+2}$ ions. Our observation of a continuous evolution in the temperature dependent magnetic susceptibility with doping level $x$ in the similar superconducting $La_{2-x}Sr_xCuO_4$ system [118] and inelastic magnetic neutron scattering investigations on this system [L4] indicated that the Cu local moments survive the insulator to superconducting metal transition with increasing $x$. The undoped parent compounds of subsequently studied families of layered cuprate superconductors were also found to become antiferromagnetic, as illustrated by the discoveries of antiferromagnetism by the PI and collaborators in $YBa_2Cu_3O_6$ [99, 106], $Ca_{0.85}Sr_{0.15}CuO_2$ [122] and $Sr_2CuO_2Cl_2$ [128]. (For an extensive review of the magnetic properties of the single-layer cuprates, see [O23]). These observations indicated that the cuprate high $T_c$ superconductors should be viewed as doped antiferromagnetic insulators. This situation was unprecedented in the history of superconductivity, where superconductivity was previously precluded by the presence of a dense array of local magnetic moments on the same sublattice of the structure on which the superconductivity would have resided. This history in turn suggested that a different mechanism from the conventional electron-phonon interaction may be responsible for the high $T_c$ superconductivity observed in the cuprates. A leading candidate, following the above observations, has been an electronic mechanism involving Cu spin interactions. However, there is still no consensus on the mechanism, so it is difficult to predict where to look for the next high $T_c$ superconductor system or family. The main thrust of the present work is to attempt to synthesize new oxide materials in the search for novel behaviors and ground states, including high temperature superconductivity. The ultimate goal in the latter context is to synthesize a superconducting system with a transition temperature above room temperature, necessarily leading to new physics and hopefully allowing superconductivity to become beneficial and ubiquitous in power transmission, electronic devices, and generally in our everyday lives.

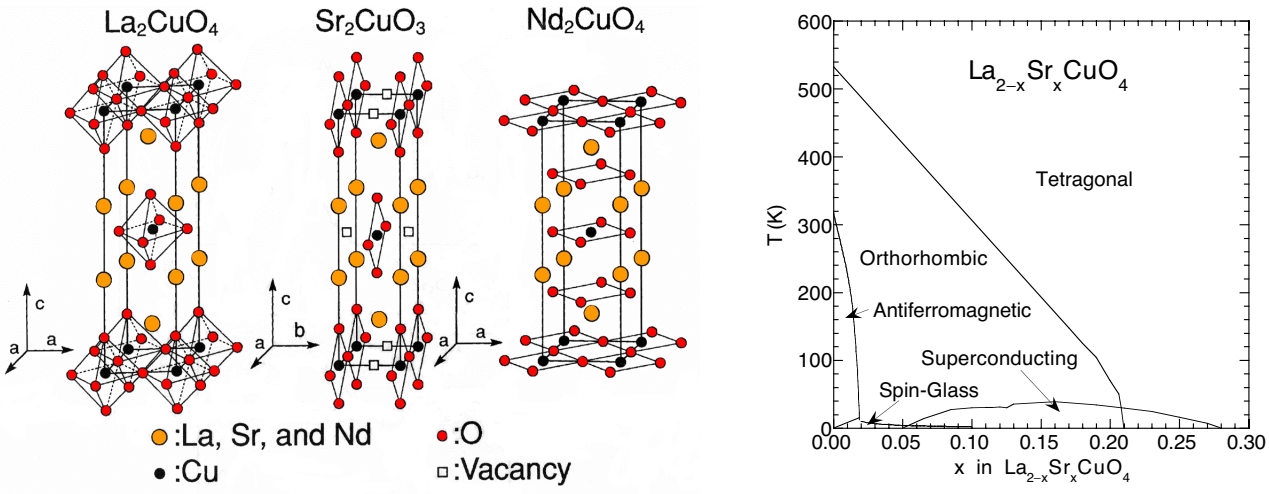



Fig. 1 (color) (Left figure) Comparison between the body-centered tetragonal $K_2NiF_4$ (*I4/mmm*) crystal structure of $La_2CuO_4$ (above 530 K, called the T structure), the orthorhombic (*Immm*) crystal structure of $Sr_2CuO_3$, and the body-centered tetragonal (*I4/mmm*) crystal structure of $Nd_2CuO_4$ (called the T' structure).

Fig. 2 (Right figure) Structural, magnetic and superconducting phase diagram of the $La_{2-x}Sr_xCuO_4$ system [O23].

In addition to substitutional doping of the insulating cuprate parent compounds as in the $La_{2-x}(Ca,Sr,Ba)_xCuO_4$ systems, anionic doping, usually involving oxygen addition or intercalation, has been utilized to produce superconducting samples. The most important example is the $YBa_2Cu_3O_{6+x}$ ($0 \leq x \leq 1$) system noted above where hole-doping is achieved for $x > 0$ and superconductivity is observed for x larger than about 0.4 [O12, O13]. The optimum $T_c$ of around 93 K is achieved for $x$ close to 1. In a related vein, we discovered new phases that were induced by hydrogen reduction and by subsequent oxidation of $Ln_2CuO_4$ compounds (Ln = La, Pr, Nd, Sm, Eu, Gd) [132]. Another important example of an oxygen-doped system is the $La_2CuO_{4+x}$ ($0 \leq x \leq 0.12$) system where $T_c$ up to about 45 K is attained for the larger x values. We have synthesized large homogeneous polycrystalline [150] and single crystal [158] samples using room-temperature electrochemical oxidation of $La_2CuO_4$ in aqueous base. In a collaboration with J. D. Jorgensen's group at Argonne National Laboratory who carried out structural studies using powder neutron diffraction, we found that the excess oxygen occupies empty sites between the $CuO_2$ layers [153] which are in fact completely occupied in the related $Nd_2CuO_4$ structure shown in the right-hand panel of Fig. 1. We also mapped out the structural ($T,x$) phase diagram of this system using the polycrystalline samples [162]. A miscibility gap was discovered between $x = 0.011$ and $x = 0.055$; antiferromagnetic ordering occurs below the low end and superconductivity above the high end compositions of the miscibility gap, with a two-phase mixture in between, which was confirmed by subsequent single crystal neutron diffraction studies [160]. This phase diagram is distinctly different from that of the $La_{2-x}Sr_xCuO_4$ system (Fig. 2; for a review, see [O23]), which has no such miscibility gap versus doping level $x$. We have also determined the temperature dependence of the oxygen diffusion rate in $La_2CuO_{4+x}$; oxygen diffusion was found to be significant to remarkably low temperatures of around 200 K [192]. R. J. Birgeneau's group at MIT later carried out neutron diffraction structural studies on one of our superconducting $La_2CuO_{4+x}$ crystals and on other crystals synthesized there [190]. They discovered that for compositions beyond

the miscibility gap, the intercalated oxygen is "staged", i.e., occupies the space only between every $n^{th}$ $CuO_2$ layer ($n$ = 4, 6, …), where the stage number $n$ depends on the excess oxygen concentration $x$.

An important and interesting aspect of the hole doped high $T_c$ cuprates is that the doped holes have often been found to occur in a non-random distribution within the $CuO_2$ planes. Some of the earliest evidence for this was found in 1992-1993 from our observed scaling of the magnetic susceptibility versus temperature and doping level $x$ of the lightly-doped $La_{2-x}Sr_xCuO_4$ system, where the doped holes were inferred to segregate into thin one-dimensional walls separating undoped, locally antiferromagnetic domains [151]. Additional evidence for this configuration was found from $^{139}$La NQR measurements carried out in collaboration with F. Borsa's group [146, 154, 182]. Neutron diffraction evidence for the existence of static doped-hole segregation was obtained by a Brookhaven group in 1995 on the compound $La_{1.48}Nd_{0.4}Sr_{0.12}CuO_4$ [L5]. The one-dimensional doped-hole configurations separating undoped locally antiferromagnetic domains are now called "stripes". "Stripe physics" has become ubiquitous in the high $T_c$ field (for a review, see [O23]). It is not yet clear whether the occurrence of stripes helps or hinders the high temperature superconductivity in the layered cuprate systems.

As is well-known, the tetragonal $K_2NiF_4$ (T) structure compound $La_2CuO_4$ (left panel in Fig. 1) can be hole-doped but not electron-doped, whereas the tetragonal $Nd_2CuO_4$ (T') structure compounds like $Nd_2CuO_4$ (right panel in Fig. 1) can be electron-doped but not hole-doped (see [O23]). Chlorine can sometimes be incorporated into these structures. The T-structure tetragonal Cl-containing compound $Sr_2CuO_2Cl_2$ exists in which the two apical oxygen atoms in $La_2CuO_4$ are replaced by Cl and the La by Sr, leaving the $CuO_2$ layers intact. This compound remains tetragonal to low temperatures (instead of distorting to a lower symmetry structure like $La_2CuO_4$ does below 530 K [92]) and has been important in establishing the intrinsic magnetic properties of the undistorted $CuO_2$ square lattice [127, 128, 130, 131, 144, 181, 191, 204]. We have carried out experiments attempting to electron-dope T-structure $La_2CuO_4$ by partially substituting O by Cl to form $La_2CuO_{4-y}Cl_y$ (J. M. Hill and D. C. Johnston, 2003, unpublished). The solid state syntheses were carried out in a variety of ways and at various temperatures using either CuCl or LaOCl as Cl sources. Consistent with all previous work on this compound, we found that electron doping of the T structure did not occur using the described method.

The $La_{2-x}Ca_xCuO_4$ system exhibits superconductivity up to temperatures of about 20 K for $x \geq 0.10$ [L3]. The system was found to be slightly oxygen deficient [L37]. Remarkably, nothing at all is currently known about the normal state magnetic properties or magnetic phase diagram of this hole-doped system. Therefore, we began a study of the magnetic susceptibility and oxygen content of this system to determine if there are any interesting qualitative differences with those of the well-studied $La_{2-x}(Ba, Sr)_xCuO_4$ systems (J. M. Hill and D. C. Johnston, 2003-2004, unpublished). From the orthorhombicity of these orthorhombic samples, defined as 2(b-a)/(a+b), versus Ca concentration $x$, we deduce that the tetragonal-to-orthorhombic structural transition temperature $T_o$ decreases through room temperature (300 K) at a Ca concentration $x \approx 0.17$. By contrast, $T_o$ in the $La_{2-x}Sr_xCuO_4$ system decreases below room temperature at a much lower Sr concentration $x \approx 0.10$ (see [O23]). The oxygen contents of our samples were determined by reduction under hydrogen in a Perkin-Elmer thermal gravimetric analyzer. There is no indication that the samples are oxygen deficient to within the error of ± 0.04 in the formula $La_{2-x}Ca_xCuO_4$, contrary to the results in [L37]. The magnetic susceptibility, however, is a much more sensitive measure of the oxygen content. $La_2CuO_4$ samples synthesized in air generally exhibit both a reduced Néel temperature $T_N$ and a tiny volume fraction of superconductivity due to the absorption of excess oxygen from the air during synthesis. This indicates that the amount of excess oxygen in the $La_2CuO_{4+x}$ samples is just within the miscibility gap between antiferromangetic and superconducting compositions for $0.011 \leq x \leq 0.055$ as discussed in the previous section. From magnetic susceptibility measurements, the samples show both antiferromagnetic

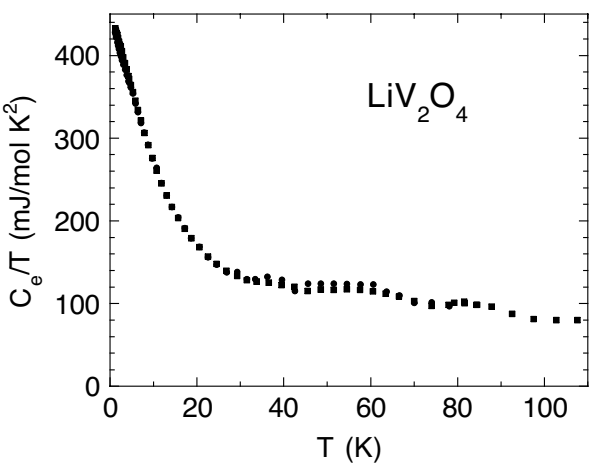
ordering at the peaks of the susceptibility and a trace of superconductivity (< 0.1 vol %) below about 40 K evidently due to excess oxygen absorbed from the air during synthesis. Studies of $La_{2-x}Sr_xCuO_4$ showed a systematic decrease of $T_N$ followed by the disappearance of long-range antiferromagnetic order at $x \approx 0.02$ (see [O23]). A similar smooth systematic decrease with increasing $x$ does not occur for $La_{2-x}Ca_xCuO_4$, perhaps due to differences in the oxygen contents of the various samples. Experiments are in progress to remove the excess oxygen from the whole series of samples by annealing them in an inert gas and to extend our studies to higher Ca concentrations.

Heavy Fermion Spinel Compound $LiV_2O_4$ and Related Materials

An important question regarding the origin of high temperature superconductivity in the layered cuprates is: what is so special about copper? One of our strategies for researching this question and to search for additional novel physics is to examine systems in which the spin $S = 1/2$ $d^9$ $Cu^{+2}$ ion is replaced by 3d transition metal cations with the same spin $S = 1/2$ but with a $d^1$ configuration instead, so that now there is one electron in the 3d shell instead of one hole. An ideal candidate for this purpose is vanadium, which shows stable oxidation states in oxides of +2, +3, +4 (especially), and +5. The cation $V^{+4}$ has $S = 1/2$ with one electron in the $d$ shell. Our initial studies were of the mixed perovskite system $LaVO_3$-$SrVO_3$. We hoped to see novel physics emerge near the known composition-induced metal-insulator transition, which was not realized [147, 148].

We then moved on to look at the enigmatic compound $LiV_2O_4$ with the fcc normal spinel structure, where the vanadium cations formally have a $d^{1.5}$ configuration [O17]. Due to the non-integral oxidation state of the V cations and their crystallographic equivalence, symmetry demands that the compound be metallic, which in fact was found long ago from resistivity measurements on small hydrothermally grown single crystals [L6]. Despite the metallic character, the magnetic susceptibility was known to show a Curie-Weiss behavior down to low temperatures (4 K) corresponding to a local magnetic moment system with $S = 1/2$ and spectroscopic splitting factor $g$ close to 2 [L7]. This behavior suggested that one of the 1.5 $d$-electrons per vanadium was somehow localized on each V atom and the remaining 0.5 $d$-electron per vanadium was itinerant and responsible for the metallic character. The Weiss temperature in the Curie-Weiss behavior was around –60 K, and yet long range antiferromagnetic ordering was not observed above 4 K. In fact, the V sublattice of the structure consists of corner-sharing tetrahedra, themselves consisting of V triangles, so the V sublattice is strongly geometrically frustrated for antiferromagnetic ordering and the possibility therefore existed that long-range ordering would be completely precluded. If so, how would the spin entropy of the system be dissipated upon cooling? Is new physics involved?

This question led us to an intensive investigation of the synthesis, characterization, and properties of high-purity $LiV_2O_4$ which resulted in our discovery in 1997 of this material as the first clear example of a $d$-electron heavy fermion system as reflected in the structural, magnetic susceptibility, specific heat, muon spin relaxation, and NMR properties below about 25 K (the "Kondo" or "coherence" temperature $T_K$) [205]. Thus the answer to the motivating question posed above is that the spin entropy of the presumed V local moments is converted on cooling below $T_K$ to entropy of the conduction carriers which is manifested as a huge (0.44 J/mol $K^2$) zero temperature electronic specific heat coefficient as shown in Fig. 3. As the heavy fermion state develops below $T_K$, the local moment behavior of the V sublattice is transformed to large nearly temperature-independent Pauli paramagnetism as shown in Fig. 4 (the Curie-Weiss behavior below 25 K previously reported by others was due to a contribution from magnetic defects and/or impurities). The heavy fermion behavior of $LiV_2O_4$ was completely unexpected by the condensed matter physics community since the extended nature of the magnetic V $d$-orbitals (compared to $f$-orbitals in $f$-electron heavy fermion compounds) and their expected strong hybridization with oxygen 2$p$ orbitals would seem to preclude such behavior. The presence of potentially new physics was indicated.



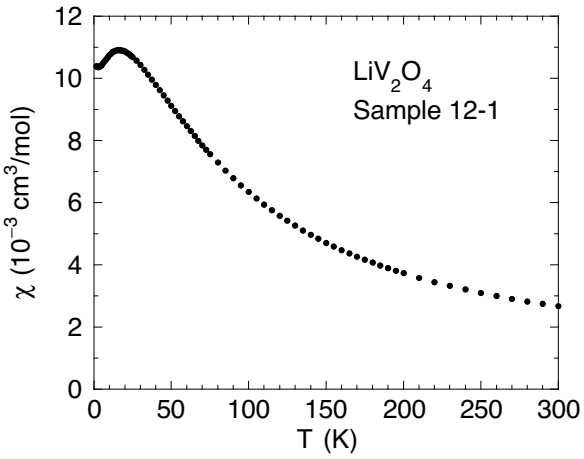




Fig. 3 (Left figure) Electronic specific heat $C_e$ divided by temperature $T$ versus $T$ for high purity $LiV_2O_4$. The lattice contribution has been subtracted. The extrapolated zero temperature limit of $C_e/T$ is 0.44 J/mol $K^2$.

Fig. 4 (Right figure) Magnetic susceptibility _ versus temperature $T$ for high-purity $LiV_2O_4$. The weak broad maximum occurs at 16 K. A Curie-Weiss behavior occurs above about 50 K.

Subsequent additional measurements of the above properties and also of the thermal expansion were carried out over the next few years which all self-consistently supported our original heavy fermion interpretation for the low-temperature properties of $LiV_2O_4$ [207, 210, 216, 217, 224]. In addition, electrical resistivity measurements down to 0.3 K on small hydrothermally grown single crystals by a group in Japan showed a $T^2$ dependence as expected for a Fermi liquid, with an extremely large coefficient consistent with a very heavy Fermi liquid [L8-L10]. Their specific heat, magnetic susceptibility, $^7$Li NMR, and thermal expansion measurements on the crystals [L8-L10] were all consistent with our previous results on polycrystalline samples. They also found that the Hall coefficient shows a distinct peak at about 15 K and crosses over from hole-like to electron-like on heating above 50 K [L8, L10]. Two inelastic magnetic neutron scattering studies have been carried out on polycrystalline samples [L11-L14]. Both studies found behaviors at low temperatures consistent with those known for *f*-electron heavy fermion compounds. They disagreed, however, on the nature of the magnetic state above $T_K$. Krimmel et al. [L11-L13] characterized this behavior as that of a nearly *ferromagnetic* metal, whereas the more complete later data of Lee et al. indicated that the heavy fermion state develops on cooling from a state with short-range *antiferromagnetic* spin correlations as expected from the antiferromagnetic Weiss temperature in the Curie-Weiss magnetic susceptibility behavior above $T_K$ [L14]. Inelastic magnetic neutron scattering studies on large single crystals are clearly called for to provide more complete information on the generalized susceptibility and magnetic excitations both below and above $T_K$. Crystals of the required minimum size (at least 1 g) have not been synthesized up to now. A plethora of theories have been advanced to explain the mechanism for formation of the heavy fermion mass in $LiV_2O_4$ below $T_K$, but there is as yet no consensus on the correct approach (for a brief review of some of the theories, see [224]).

Many experiments of various types on high purity polycrystalline and single crystal samples of the metallic transition metal spinel-structure oxide compound $LiV_2O_4$ showed heavy fermion behaviors from 0.3 K to around 20 K as just described. However, claims have appeared in the literature [L15-L17] that a different behavior occurs in the $^7$Li NMR spin-lattice relaxation rate (and other properties) below 1 K from the heavy fermion behavior we observed in this quantity above 1 K. In addition, they

found a non-exponential ("stretched exponential") decay to the nuclear magnetization in their pulsed NMR experiments, whereas we observed and reported a strictly exponential decay. To address these claims and understand the origin of their divergent results, we extended our $^7$Li NMR measurements of this compound down to 0.5 K (S.-H. Baek. F. Borsa, S. Kondo, and D. C. Johnston, unpublished work). We found the temperature dependence of the nuclear spin-lattice relaxation rate to be consistent with our previous results for a high-purity sample with an equivalent $S = 1/2$ magnetic defect concentration of about $n = 0.01$ mol%. In addition, we found that a sample with a higher magnetic defect concentration $n = 0.3$ mol% shows a distinctly different behavior that is similar to that of Refs. [L15-L17]. Finally, we found a strictly exponential decay of the nuclear magnetization for the high purity sample but which becomes non-exponential as the magnetic defect concentration increases. In fact, we had already shown in 1997 from our early μSR studies of two samples with different magnetic defect concentrations that static spin glass ordering occurs below 0.8 K when $n \sim 0.3\%$, whereas no static spin ordering occurs above 0.02 K for our highest purity sample with $n \sim 0.01\%$ [205]. These results indicate that (i) the ground state of high purity $LiV_2O_4$ is a heavy fermion ground state with heavy fermion behaviors at low temperatures as we originally reported, (ii) the ground state of $LiV_2O_4$ is qualitatively changed from a heavy fermion ground state to some type of spin-glass state as the concentration of magnetic defects in the material increases above about 0.1 mol%, and (iii) the authors of [L15-L17] carried out their experiments on samples with high magnetic defect concentrations and did not measure the intrinsic properties of pure $LiV_2O_4$.

As an extension of our previous work on polycrystalline samples of $LiV_2O_4$, in preliminary experiments we attempted to grow single crystals of this compound using fused salt electrolysis of molten electrolytes with composition $(Li_2O)_xV_2O_5$ with $x = 0.5$-1.0, in 50 mL Pt crucibles under Ar atmosphere at 600-650 ˚C with currents of 0.1-10 mA. This technique instead consistently yielded single crystals of the known compound $LiV_2O_5$, as determined from single crystal x-ray diffraction measurements, with dimensions up to 1x2x15 mm$^3$. Single crystals of this material had been grown before using flux growth as reported in the literature, but our crystal growth method as applied to this compound is new.

Of the hundreds of known spinel structure transition metal oxide compounds, only two remain metallic to low temperatures: $LiV_2O_4$ and $LiTi_2O_4$. Superconductivity was discovered in 1973 by the PI at temperatures up to 13.7 K in $LiTi_2O_4$ [5, 12, 13]. Until the advent of high-$T_c$ cuprates in 1986, this was the highest $T_c$ oxide compound. The cation-substituted Ti spinel systems $Li[Li_xTi_{2-x}]O_4$ and $Li[Al_xTi_{2-x}]O_4$ undergo composition-induced metal-to-insulator transitions as x increases. In collaboration with R. J. Gooding's group at Queen's University, we studied these transitions using a quantum site percolation model [230]. The results suggested that strong electronic correlations are both present and important in these Ti oxide spinel systems, just as they obviously are in $LiV_2O_4$.

Pyrochlores

We have extended our studies of the fcc spinel compound $LiV_2O_4$ to include synthesis and properties of pyrochlore structure materials of general composition $A_2B_2O_6O'$, where A is usually a lanthanide and B a transition metal. The O and O' atoms occupy crystallographically distinct lattice sites. Both the A and B sublattices are identical to the V sublattice in $LiV_2O_4$. However, the A atoms are 8-fold coordinated by O whereas the B atoms have 6-fold octahedral coordination by O. For antiferromagnetically coupled B atoms, the B sublattice is geometrically frustrated for long-range antiferromagnetic ordering, as in $LiV_2O_4$. Hypothetical metallic members of this structure class containing local magnetic moments therefore have the potential to exhibit heavy fermion behavior and thus be included in our new class of *d*-electron heavy fermion compounds. The known pyrochlore compound $Lu_2V_2O_7$ [L18, L19] contains $d^1$ $S = 1/2$ $V^{+4}$ cations and is a ferromagnetic n-type

semiconductor with a Curie temperature $T_C$ = 73 K and an activation energy of 0.2 eV [L19-L23]. We carried out a clean synthesis of hypothetical $Lu_2V_2O_2F$ (where F replaces O') in sealed molybdenum tubes at 1250˚C, which would formally contain $d^{1.5}$ V cations as in $LiV_2O_4$ and would be metallic if it formed. Unfortunately, the compound did not form (G. Knoke, J. M. Hill, and D. C. Johnston, 2002, unpublished).

We discovered a new series $Lu_2V_2O_{7-x}$ of oxygen deficient pyrochlores with $0.4 \leq x \leq 0.7$ by annealing $Lu_2V_2O_7$ in flowing $H_2$ (G. Knoke, J. M. Hill, and D. C. Johnston, unpublished work). Magnetization measurements showed that $T_C$ for the oxygen-deficient materials is much lower than the value of 73 K for the $Lu_2V_2O_7$ parent compound, reaching 15 K for $x = 0.7$. Above $T_C$, the magnetic susceptibility follows the Curie-Weiss law with a Curie constant consistent with a mixture of localized spin $S = 1/2$ $V^{+4}$ and $S = 1$ $V^{+3}$ cations dictated by the oxygen deficiency $x$ in an ionic model. Although we could not carry out electronic transport measurements due to the powder nature of these samples, from the composition dependence of the Curie constant we infer that the series does not become metallic within the composition range investigated. X-ray crystallographic studies demonstrated that the oxygen deficiency is accompanied by Lu-V antisite disorder, which is reflected in a strong reduction in the intensities of the x-ray reflections with (odd odd odd) Miller indices. An outstanding question is why the Curie temperature decreases with increasing oxygen deficiency, even though the average spin of the vanadium sublattice increases with oxygen deficiency. The explanation may have its origins in the antisite disorder just mentioned and deserves further study.

Spin Chain Oxide Compounds

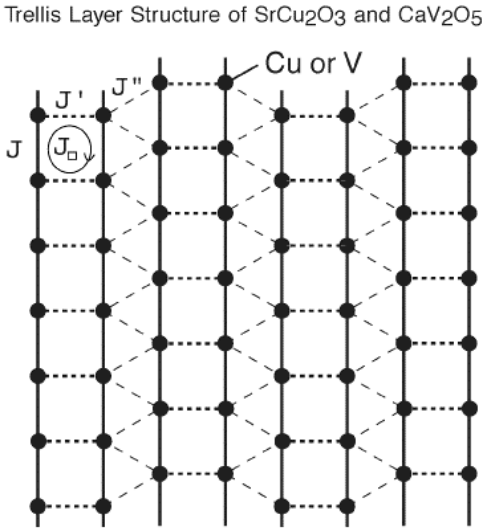
The field of low-dimensional magnetism received a major boost following the discovery of the cuprate high $T_c$ superconductors and of the magnetic character of the parent and doped compounds discussed above. For example, it was discovered theoretically in 1988 that the ground state of the spin $S = 1/2$ square lattice Heisenberg antiferromagnet has long-range antiferromagnetic order (but only at temperature $T = 0$ for an isolated layer) [L23, L24]. A surprising finding from conformal field theory in 1994 was that the magnetic susceptibility of the fiducial S = 1/2 Heisenberg chain initially increases with temperature from $T = 0$ with infinite slope, arising from a low-temperature "logarithmic correction" to the susceptibility of the form 1/log(T*/T), where T* is a characteristic temperature [L25]. Our numerical calculations support this finding and in addition yield the next few higher order logarithmic correction terms [222, 223]. We also carried out very high accuracy calculations and simulations of the magnetic susceptibility versus temperature of both the uniform antiferromagnetic $S = 1/2$ Heisenberg chain and the alternating exchange chain in which the antiferromagnetic exchange interaction between adjacent spins alternates between two values $J_1$ and $J_2$. We obtained a high-accuracy two-dimensional function describing the susceptibility versus temperature for $0 \leq J_1/J_2 \leq$ infinity by fitting the numerical data to within the accuracy of the data [222]. This high-accuracy function will be of great use in the future for fitting experimental susceptibility data for uniform and alternating-exchange $S = 1/2$ chain compounds (see the fits of the susceptibility of $Sr_2CuO_3$ below and of $(VO)_2P_2O_7$ in the next section). The new high-accuracy numerical results on the magnetic susceptibility versus temperature of the uniform chain [L25, 222, 223] supplant the venerable Bonner-Fisher calculations from 1964 [L26], where the prediction is too low by as much as 10% at the lower temperatures (see Fig. 8.1 in [O23]). For reviews of these and other theoretical developments on the antiferromagnetically coupled S = 1/2 spin chain and square lattice, see [O23] and [222].

On the experimental side, in 1995 we discovered that the linear chain compound $Sr_2CuO_3$ is a $S = 1/2$ Heisenberg chain with a very large Cu-Cu exchange coupling $J \sim 2200$ K [173, 203]. The structure of this compound (middle panel of Fig. 1) is derived from that of the tetragonal high temperature $K_2NiF_4$ structure of $La_2CuO_4$ (left panel of Fig. 1) by removing entire lines of O atoms

within the $CuO_2$ layers along one of the (100) directions and substituting Sr for La, resulting in an orthorhombic unit cell. From our fits to the magnetic susceptibility versus temperature, the nearest-neighbor antiferromagnetic exchange coupling strength between the $S = 1/2$ copper spins is $J = 2150(150)$ K [173, 203]. This $J$ value is about 50% larger than in the above layered cuprate parent compounds (~1500-1600 K, see [O23]), even though the Cu-O-Cu bonding is nearly identical in the two cases (we address this disparity below). Long-range antiferromagnetic order due to interchain coupling does not set in until the temperature decreases to $T_N = 5.4$ K [L19], so the ratio $T_N/J = 0.002$ is very small. The extrapolated zero-temperature ordered moment from magnetic neutron diffraction measurements on single crystals is only 0.06(3) $\mu_B$/Cu [L27]. The expected saturation moment is $\mu_{sat} = gS\mu_B = 1.1$ $\mu_B$/Cu, and the much lower value observed was attributed to quantum fluctuations associated with the quasi-one-dimensionality of the spin system [L27]. These properties together establish $Sr_2CuO_3$ as one of the best model $S = 1/2$ quasi-one-dimensional antiferromagnetic Heisenberg spin chain systems to date. Up to now there are no reports of doping this compound into the metallic state. As is well-known, the physics of one-dimensional antiferromagnets is quite different in certain respects from higher-dimensional systems. We are very interested in following the evolution in the physical properties of one-dimensional antiferromagnets such as linear chains and spin ladders (below) with doping to compare and contrast the results with the extensive results on the two-dimensional layered cuprates. We anticipate that the nature of the evolution may be quite different than in higher-dimensional systems and that new physics might be accessible via this route.

We tried to dope the quasi-1D spin-1/2 Heisenberg chain compound $Sr_2CuO_3$ using electrochemical oxidation in aqueous base solution (J. M. Hill and D. C. Johnston, unpublished work). As described above, we successfully used this technique previously to electrochemically dope holes into insulating quasi-2D $La_2CuO_4$ to produce superconducting $La_2CuO_{4+y}$ ($0.06 \leq y \leq 0.12$) powders and crystals. We found, however, that $Sr_2CuO_3$ decomposes when exposed to either water vapor or liquid water, the main decomposition product being $Sr_2Cu(OH)_6$. The structure of this compound [L28] consists of isolated $Cu(OH)_6$ octahedra. We carried out a thorough study of the EPR, magnetization versus magnetic field at fixed temperatures $T$ and magnetic susceptibility $\chi$ versus $T$ of $Sr_2Cu(OH)_6$ [228]. The $\chi(T)$ data are well described by a Curie-Weiss law, with a small Weiss temperature $\theta \approx -3$ K that corresponds to a weak antiferromagnetic Cu-Cu superexchange interaction $J \approx 1$ K. The formation of $Sr_2Cu(OH)_6$ on the surfaces of $Sr_2CuO_3$ samples exposed to air is therefore likely to be the previously unknown source of the variable amounts of Curie-like contributions to the susceptibility of $Sr_2CuO_3$ previously documented by us [173]. $Sr_2Cu(OH)_6$ also serves as a nice reference material for comparison with the magnetic properties of the much more strongly interacting ($J \sim 2000$ K) high-$T_c$ cuprates. We then tried to dope holes into $Sr_2CuO_3$ via electrochemical oxidation in a nonaqueous electrolyte consisting of tetramethylammonium hydroxide in methyl alcohol, a technique reported to be successful for oxidizing $La_2CuO_4$ [L29], but no evidence that doping occurred was found from either crystallographic or magnetic susceptibility measurements.

The ambient pressure (AP) phase of the compound vanadyl pyrophosphate, $(VO)_2P_2O_7$, contains spin-1/2 $d^1$ $V^{+4}$ ions. The PI and collaborators discovered in 1987 from magnetic susceptibility data that this compound exhibits a spin gap and modeled the magnetic susceptibility versus temperature data in terms of an antiferromagnetic alternating-exchange spin chain model [90]. For a review of the subsequent convoluted history of the study and interpretation of the magnetic properties of this compound, which for a time was considered to be a spin ladder compound, see [227]. Ultimately our original model that we used to fit the susceptibility data now appears to be the correct one. In collaboration with experimentalists in Japan, we have measured and modeled new magnetic susceptibility versus temperature data for both polycrystalline and single crystal samples of this compound, and also of a new high-pressure (HP) modification of this compound discovered in 1999



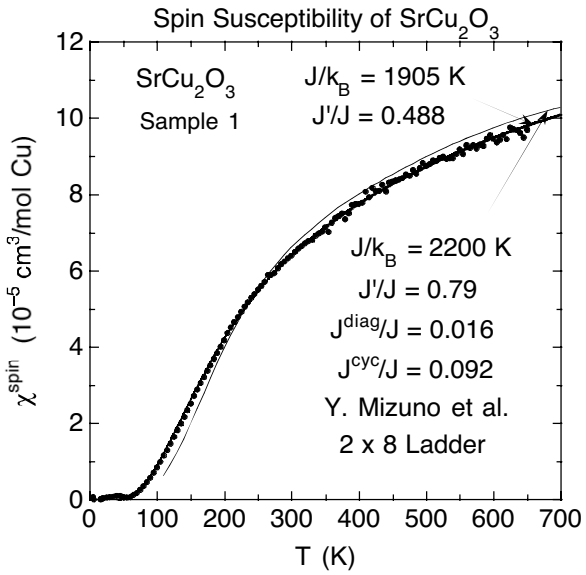




Fig. 5 (Left figure) The "trellis layer" structure of the Cu and V atoms in $SrCu_2O_3$ and $CaV_2O_5$. Within each two-leg ladder, an oxygen atom is situated between each nearest-neighbor pair of transition metal atoms.

Fig. 6 (Right figure) Measured spin susceptibility of $SrCu_2O_3$ (dots), together with our fit (solid curve) on the basis of the nearest-neighbor Heisenberg model. Also shown is an ab initio calculation [35] in which a four-spin cyclic exchange interaction and a weak diagonal antiferromagnetic interaction is included in addtion to the principal Heisenberg exchange interaction. The exchange interactions for each fit are shown in the figure.

[225], to determine the magnetic exchange interactions between the V spins [225, 227]. To do these fits, the PI used his new high-accuracy two-dimensional function for the susceptibility versus temperature and $J_1/J_2$ of $S = 1/2$ uniform and alternating-exchange Heisenberg chains discussed above. In a *tour de force* using this function to fit the experimental data, we found that the AP phase contains *two* distinct types of alternating-exchange $V^{+4}$ chains, each with its own spin gap and pair of alternating antiferromagnetic exchange interactions. This result is consistent with the crystal structure, which indicates the presence of two distinguishable spin chains, and with the magnon dispersion relations found from previous inelastic neutron scattering measurements [L30]. In addition, the new HP phase was found to contain a single type of $V^{+4}$ $S = 1/2$ alternating-exchange chain, again consistent with that crystal structure.

Spin Ladder Oxide Compounds

Another offshoot of the high $T_c$ field was spin ladder physics which began in earnest as a subfield of low-dimensional magnetism in 1993 (for reviews, see [O23] and [230A]). An $n$-leg spin ladder is a spin configuration in which the spins lie on the vertices where the $n$ legs (or rails) meet the perpendicular rungs of the ladder. A square spin lattice, as in the layered high $T_c$ cuprates, is one limiting case with $n$ = infinity, and a linear spin chain is the other limit with $n = 1$. An early theoretical prediction was that $S = 1/2$ ladders with even $n$ should have an energy gap ("spin gap") between a (nonmagnetic) singlet ground state and the lowest-lying (magnetic) triplet excited states, whereas ladders with odd $n$ should be gapless [e.g., L31, L32]. This prediction was confirmed via magnetic susceptibility and other measurements on the $S = 1/2$ spin ladder compounds $SrCu_2O_3$ with $n = 2$ and

$Sr_2Cu_3O_5$ with $n$ = 3 [L33] (and of course on the above $Sr_2CuO_3$ with $n$ = 1), all of which are insulators. The "trellis layer" structure of $SrCu_2O_3$ is shown in Fig. 5; the two-leg ladder compound $CaV_2O_5$ has a similar structure within the $V_2O_3$ planes. The 180˚ Cu-O-Cu superexchange path within a ladder is strong and antiferromagnetic, whereas the 90˚ Cu-O-Cu path between ladders is weakly ferromagnetic and is frustrating, leading to an effective magnetic isolation of the ladders from each other. From the crystal chemistry of these materials, one expects that the Cu-Cu superexchange coupling should be about the same in all three cuprate compounds, with the rung ($J$') and leg ($J$) couplings about the same with $J'/J \sim 1$ [L31, L32]. However, in 1996 we discovered that the (rather sparse) existing magnetic susceptibility versus temperature calculations *based on the nearest-neighbor Heisenberg model* for the $S$ = 1/2 two-leg ladder, when compared with the experimental magnetic susceptibility data for $SrCu_2O_3$, indicated instead that $J'/J \sim 0.5$, with $J \sim 2000$ K [198]. This $J$ value is on the order of that in the linear chain compound $Sr_2CuO_3$ but larger than the values of 1500-1600 K previously inferred for the square lattice cuprate parent compounds [O23]. However, the *average* of $J$' and $J$ is about 1500 K. These conundrums were further addressed and confirmed both theoretically and experimentally by the PI and collaborators in very extensive work on spin ladders described next.

We recently completed an extensive five-year combined theoretical and experimental study, in collaboration with scientists in Germany, Switzerland, Russia, and Japan, of the magnetic susceptibility versus temperature $\chi(T)$ of insulating spin $S$ = 1/2 two- and three-leg Heisenberg ladders and ladder oxide compounds [230A]. The $\chi(T)$ of isolated two-leg ladders with spatially anisotropic antiferromagnetic (AF) Heisenberg exchange was calculated by quantum Monte Carlo (QMC) simulations, with and without ferromagnetic (FM) second-neighbor diagonal intraladder coupling, and for two-leg ladders coupled into a two-dimensional (2D) stacked ladder configuration or a 3D $LaCuO_{2.5}$-type interladder coupling configuration. We obtained accurate analytical fits and interpolations of these data and of previously reported related QMC $\chi(T)$ simulation data for the isolated ladder with spatially isotropic exchange, for the 2D trellis layer configuration and for isotropic and anisotropic three-leg ladders. We also calculated the one- and two-magnon dispersion relations for the isolated 2 x 12 ladder with $0.5 \leq J'/J \leq 1$, where $J$ is the AF coupling constant along the legs and $J'$ is that along the rungs. The exchange constants in the two-leg ladder compound $SrCu_2O_3$ were estimated from LDA+U calculations. On the experimental side, we determined the detailed crystal structures of $SrCu_2O_3$ and of the three-leg ladder compound $Sr_2Cu_3O_5$. New experimental $\chi(T)$ data were obtained for $SrCu_2O_3$ and $LaCuO_{2.5}$, and for the nominally two-leg ladder vanadites $CaV_2O_5$ and $MgV_2O_5$ which are structurally similar to $SrCu_2O_3$. These and literature $\chi(T)$ data for these compounds and for $Sr_2Cu_3O_5$ were modeled using our QMC simulation fits. $CaV_2O_5$ was found to be essentially a dimer compound with AF intradimer coupling 669(3) K ($g$ = 1.96), in agreement with previous results [L34]. The leg and rung exchange constants found for isostructural $MgV_2O_5$ are very different from those in $CaV_2O_5$, as predicted previously from LDA+U calculations. For $SrCu_2O_3$, we found that $J'/J$ = 0.5 and $J$ = 1900 K, assuming a spectroscopic splitting factor $g$ = 2.1 (see the fit in Fig. 6), confirming our previous 1996 modeling results [198]. The interladder coupling $J'''/J$ = 0.01(1) perpendicular to the ladder layers was found to be very weak and on the spin-gapped side of the quantum critical point (QCP) at $J_{QCP}'''/J = 0.048(2)$ for $J'/J = 0.5$. The three-leg ladder compound $Sr_2Cu_3O_5$ was also found to exhibit strong intraladder exchange anisotropy, with $J'/J = 0.66(5)$ and $J$ = 1810(150) K for $g = 2.1(1)$. The $\chi(T)$ data for $LaCuO_{2.5}$ were consistent with $J'/J = 0.5$ with $J$ = 1700 K, again assuming $g$ = 2.1, and with a 3D FM interladder coupling $J^{3D}/J = -0.05$ which is close to and on the AF ordered side of the QCP at $J_{QCP}{}^{3D}/J = -0.036(1)$ for $J'/J = 0.5$, consistent with the observed AF-ordered ground state.

The surprisingly strong spatial anisotropy of the bilinear exchange constants within the cuprate spin ladders, $J'/J \sim 0.5$, derived unambiguously from our fits of the experimental $\chi(T)$ data by precise predictions of the Heisenberg model as described above, is in conflict with expectation from crystal chemical arguments which predict that $J'/J \geq 1$. The resolution of this conundrum is likely that the Heisenberg Hamiltonian is insufficient to describe the magnetism of the cuprate ladder compounds. In particular, we suggested that a four-spin cyclic exchange interaction is also present which has a strong influence on $\chi(T)$. This hypothesis is supported by calculations of Mizuno, Tohyama, and Maekawa that indicate the presence of this interaction in $SrCu_2O_3$ [L35]. They found that a relatively small amount (9%) of this interaction relative to $J$ has a profound influence on $\chi(T)$ and that the observed $\chi(T)$ is well reproduced by including both the Heisenberg and cyclic exchange interactions, as shown previously in Fig. 6. One important implication is that the latter interaction is also important to the magnetic properties of the square lattice cuprates. This is consistent with the conclusion of, e.g., Coldea et al. who found that this term must be included in the Hamiltonian in their fits to their inelastic magnetic neutron scattering measurements of the magnon dispersion relations in $La_2CuO_4$ [L36]. Another important implication is that the exchange interaction $J$ inferred for the square lattice and spin chain cuprates on the basis of the nearest-neighbor Heisenberg model alone is only an *effective* interaction, which is too small relative to the actual value and includes the influence of the cyclic exchange interaction (see the exchange parameters in Fig. 6). This explains why the $J$ in the linear chain cuprate $Sr_2CuO_3$ (2200 K), in which a cyclic exchange interaction cannot be present, is significantly larger than the (effective) $J$ of 1500-1600 K inferred previously (see [O23]) for the square lattice cuprates.

The Layered Orthogonal Dimer Compound $SrCu_2(BO_3)_2$.

We have been been carrying out an extensive series of experiments (J. M. Hill, S. Sanger, M. Tillman, S. Madison, and D. C. Johnston, unpublished work) to try to dope the 2D spin dimer compound $SrCu_2(BO_3)_2$ [L38]. This insulating compound has a spin gap and is of great current experimental and theoretical interest in its own right because it is the only known physical realization [L39] of the so-called Shastry-Sutherland model [L40] for a particular layered frustrated dimer spin system, for which the magnetic ground state is known exactly. Due to the geometric frustration for antiferromagnetic ordering inherent in the structure (shown in Fig. 7) [L39, L41], this compound has qualitatively different magnetic properties [L38] and interactions [L39, L42] than in the undoped layered high-$T_c$ cuprate parent compounds which have a non-frustrated square lattice arrangement of the $Cu^{+2}$ ions (see [O23]). We thus anticipate that if $SrCu_2(BO_3)_2$ can be doped, the evolutions in the magnetic and electronic properties with doping might be qualitatively different from those known for the layered high-$T_c$ cuprates, offering the potential for interesting new physics and physical behaviors. We found that sintered pellets of the compound do not react significantly with water and therefore that aqueous chemical and electrochemical doping techniques were potentially possible. We verified literature reports [L43-L45] that $La_2CuO_4$ can be hole-doped to become superconducting by reacting it with aqueous sodium hypochlorite (NaClO) solution at room temperature for several days. We then used this technique to attempt to chemically oxidize $SrCu_2(BO_3)_2$. We found that both water and aqueous NaClO have little effect on the magnetic susceptibility of sintered pellets of $SrCu_2(BO_3)_2$ as shown in Fig. 8. Powdered samples of $SrCu_2(BO_3)_2$, however, partially decomposed to CuO and a water soluble strontium borate complex when exposed to water for several hours. The powdered samples completely decomposed to CuO and $SrCO_3$ when exposed to the NaClO solution (the $CO_2$ in the $SrCO_3$ came from the air and $CO_2$ dissolved in the water). Magnetic susceptibility data on the powder

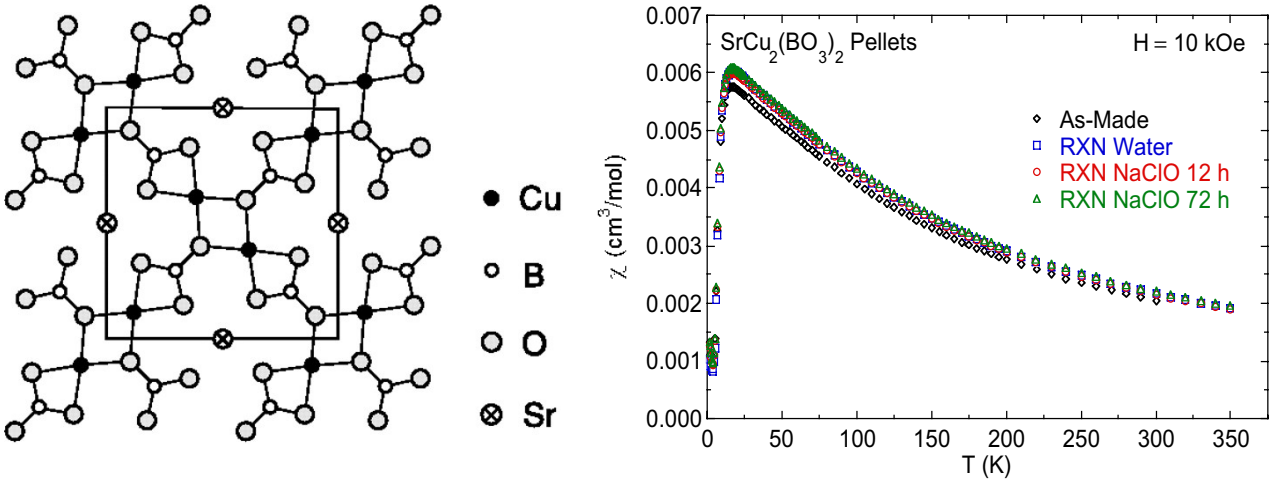



Fig. 7 (Left figure) The structure of $SrCu_2(BO_3)_2$ viewed along the *c* axis. The box shows a unit cell projected on the *ab* plane. The Sr atoms are displaced above and below the *ab* plane.

Fig. 8 (Right figure) (Color) Magnetic Susceptibility _ versus temperature *T* in an applied magnetic field of $H = 10$ kOe for pellets of as-made $SrCu_2(BO_3)_2$ (◊), $SrCu_2(BO_3)_2$ reacted with water (squares), $SrCu_2(BO_3)_2$ reacted with aqueous NaClO for 12 h (circles), and $SrCu_2(BO_3)_2$ reacted with NaClO for 72 h (triangles) [P44]. The "as-made" sample is one that received no special treatments after synthesis.

samples were both quantitatively and qualitatively different from the magnetic susceptibility of as-prepared $SrCu_2(BO_3)_2$ and consistent with the presence of a large concentration of CuO in the sample.

In addition to the aqueous chemical doping experiments discussed above, we tried to partially replace the Sr in $SrCu_2(BO_3)_2$ by La and fire the samples in air, and then planned to anneal the samples in an inert atmosphere to remove excess oxygen and thereby electron dope the material. The strategy was the same as that successfully used in the past to synthesize the electron-doped high $T_c$ cuprate $Nd_{2-x}Ce_xCuO_4$ [O23]. We found that $LaBO_3$ formed as a second phase in all samples containing La, the lattice parameters of the $SrCu_2(BO_3)_2$ were invariant with the amount of La doping, and that the magnetic susceptibility of those samples was qualitatively unchanged from the magnetic susceptibility of pure $SrCu_2(BO_3)_2$. These results all indicated that when synthesized in air, excess oxygen does not enter the lattice to form $SrCu_2(BO_3)_2O_x$, in contrast to the cuprate $Nd_{2-x}Ce_xCuO_{4+x}$ that is formed in air. Additional electron doping experiments are in progress to synthesize $Sr_{1-x}La_xCu_2(BO_3)_2$ in sealed quartz tubes. We are also planning to synthesize $Sr_{1-x-y}La_xBa_yCu_2(BO_3)_2$ in which *x* and *y* will be adjusted to keep the average (La, Ba) cation size the same as for Sr in order to reduce lattice mismatch effects. Finally, we are attempting to hole-dope $SrCu_2(BO_3)_2$ by substituting $Na^{+1}$ for $Sr^{+2}$.

<u>$MgAlB_{14}$</u>

The recent observation of superconductivity below a temperature of 39 K in the previously known compound $MgB_2$ [L46] indicated that the condensed matter physics community may have overlooked interesting physical properties in other known non-transition metal compounds. The boride compound $MgAlB_{14}$ was discovered by Ames Lab scientists a few years ago to be one of the hardest materials known [L47]. The crystal structure of $MgAlB_{14}$ consists of linear (zig-zag) chains of Al (Mg), but crystallographic studies in the literature report that the metal sites are not fully occupied; the occupation of the cation sites (0.8) is such that the cations together transfer four electrons to the boron sublattice [L48-L50]. In addition to $MgAlB_{14}$, the compound $Mg_2B_{14}$ [L51] forms in the same structure as well as $MAlB_{14}$ where M = Li, Y, Tb, Dy, Ho, Er, Tm, Yb, and Lu [L49, L52-L54]. Only the $Mg_2B_{14}$ and $LiAlB_{14}$ compounds have fully occupied metal sites, again strongly indicating that the

compound formation is dependent on electronic effects. We completed a search for unusual magnetic properties in $MgAlB_{14}$ in collaboration with B. A. Cook, J. L. Harringa, and A. M. Russell of Ames Laboratory [229]. Two types of samples were synthesized and studied: 1) powder and 2) chemically substituted and unsubstituted hot pressed pellets prepared from mechanically alloyed powders. We found that synthesis of single-phase powder samples of $MgAlB_{14}$ was not possible, mostly due to the difficulty of incorporating the Al metal starting material [P48]. Magnetic susceptibility $\chi$ versus temperature $T$, magnetization $M$ versus $T$, and isothermal $M$ versus magnetic field $H$ studies of our samples were carried out to search for superconductivity or ferromagnetism in this compound. The $\chi(T)$ measurements on a powder sample revealed temperature-independent diamagnetism with a small Curie-Weiss impurity concentration equivalent to about 1 mol% of spin-1/2 ions. In contrast, $M(T)$ and $M(H)$ data on the hot pressed samples showed evidence of ferromagnetic transitions above about 330 K. Scanning electron microscopy and Auger microprobe analysis of the hot pressed samples indicated that the samples contained significant concentrations of Fe impurities. We conclude that pure $MgAlB_{14}$ is neither a superconductor nor a ferromagnet above 1.8 K and exhibits temperature-independent diamagnetism from 1.8 K up to room temperature. The ferromagnetism observed in the hot pressed samples is likely due to Fe impurities abraded from the stainless steel mills used to mix the starting materials prior to hot pressing the samples.

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